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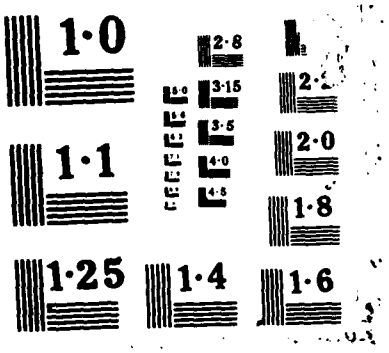
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CHARACTERIZATION OF YTTRIUM ALUMINUM GARNET /YAG/ USING 14 MeV NEUTRON ACTIVATION

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EMERGING MATERIALS DIVISION

December 1987

US ARMY LABORATORY COMMAND
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ABSTRACT

A non-destructive method for the analysis of yttrium, aluminum and oxygen in Yttrium Aluminum Garnet /YAG/ by 14 MeV neutron activation analysis had been developed and evaluated. The fast neutron induces primarily /n,2n/, /n,p/ or /n,α/ reactions with yttrium, aluminum, and oxygen to produce isotopes with measurable characteristic gamma-ray spectra. Concentrations of Y, Al and O were determined on the basis of calibrated nuclear decay emission spectra recorded on selected standards. After each analysis the crystal was returned to the furnace for further adjustments of the growth parameters, and any resultant elemental variations were observed in the next analysis. The accuracy of this method was determined by repeated analyses with various "YAG" crystals from a series of separate furnace runs.

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CHARACTERIZATION OF YTTRIUM ALUMINUM GARNET /YAG/ USING 14 MeV NEUTRON ACTIVATION

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A non-destructive method for the analysis of yttrium, aluminum and oxygen in Yttrium Aluminum Garnet /YAG/ by 14 MeV neutron activation analysis has been developed and evaluated. The fast neutron induces primarily /n,2n/, /n,p/ or /n,α/ reactions with yttrium, aluminum, and oxygen to produce isotopes with measurable characteristic gamma-ray spectra. Concentrations of Y, Al and O were determined on the basis of calibrated nuclear decay emission spectra recorded on selected standards. After each analysis the crystal was returned to the furnace for further adjustments of the growth parameters, and any resultant elemental variations were observed in the next analysis. The accuracy of this method was determined by repeated analyses with various "YAG" crystals from a series of separate furnace runs.

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INTRODUCTION

YAG /$Y_3Al_5O_{12}$/ is widely used in the solid state laser field as the light emitting source. For example, a new holographic interferometer installed at the NASA Ames Research Center is based on a frequency-doubled YAG laser[1]. Its unique physical properties, high peak power, good temporal resolution, and broad spectral coverage are critical to a variety of applications. In order to produce these high quality YAG lasers[3], it is necessary to synthesize high purity crystals with the correct stoichiometric composition. This is difficult to accomplish due to the extreme environment that is used for the synthesis and subsequent single crystal growth. In this process, yttrium oxide and aluminum oxide are mixed and then melted at temperatures above 2000 ^{o}C /Ref. 2/. It is important to find the best method to mix the oxides prior to heating to the final temperature so that large compositional variation in the crystals will not occur. The resulting crystal must be characterized in order to determine the correct chemical composition of the melt. X-ray powder diffraction[4] can be helpful in the detection of various phases present.

Accurate analysis for oxygen determination using 14 MeV neutrons has been a standard technique for many years[5]. Aluminum and yttrium each form isotopes which decay with the emission of gamma rays and can be detected and resolved with a Ge/Li/ detector.. Thus the concentration of all three elements can be determined simultaneously on the same crystal.

It is possible to use standard wet chemical techniques to analyze YAG quantitatively for aluminum and yttrium, but not oxygen[6]. Also, such analyses are destructive.

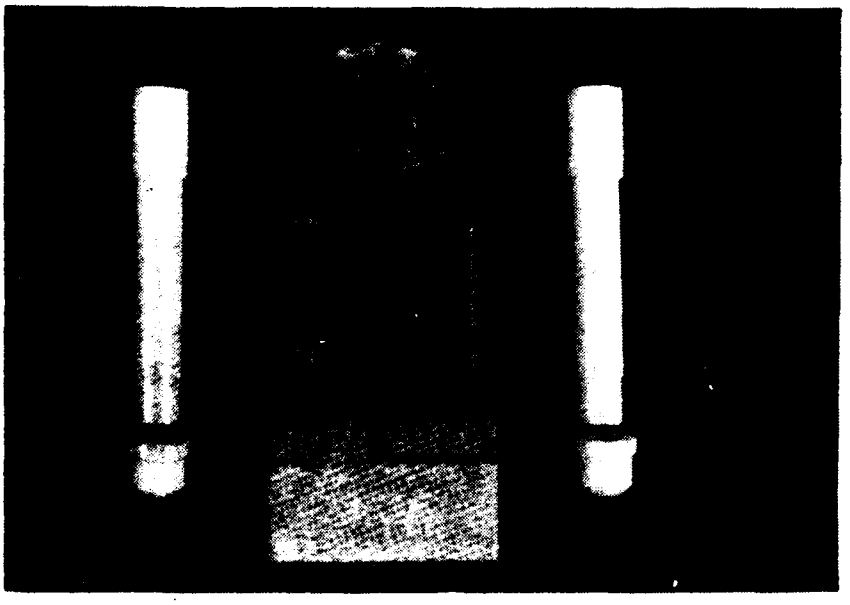

Fig. 1.

Using the 14 MeV neutron activation method, all three elements can be determined non-destructively while allowing the same analyzed crystal to be returned to the furnace for further adjustments of the growth parameters.

It is known that yttrium aluminum garnet /YAG/ can accept impurity ions into its lattice[9,10]. By judiciously selecting and incorporating optically active impurity ions, the properties of solid state materials can be tailored to specific applications.

EXPERIMENTAL

Procedure for yttrium and aluminum analysis

Samples were core drilled from the YAG boule using a 1/4" diamond core drill in Fig. 1. Sample and standard were vibrated and tamped into individual 1.250" x

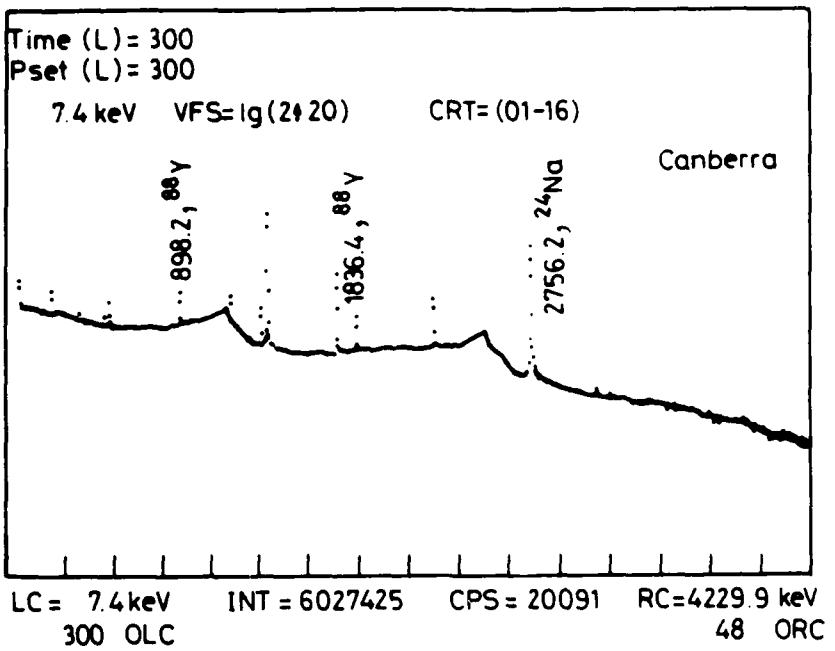

Fig. 2.

0.304" diameter polystyrene capsules. The concentrations of yttrium and aluminum were determined by counting the ^{88}Y and ^{24}Na activities produced by a 10 min irradiation using the 14 MeV neutron generator and special irradiation equipment. A Canberra Series-80 multichannel analyzer in conjunction with a high resolution lithium drifted germanium detector was used as the counting system. A typical gamma-ray spectrum of ^{88}Y, and ^{24}Na is shown in Fig. 2. By comparing the counts of the sample vs. the standard the quantitative results for yttrium and aluminum were obtained.

Procedures for oxygen analyses

The same samples used for yttrium and aluminum analyses were used for the oxygen determination. The oxygen standard used was a Lucite TM cylinder of the same dimensions as the sample. Sample and standard were placed in

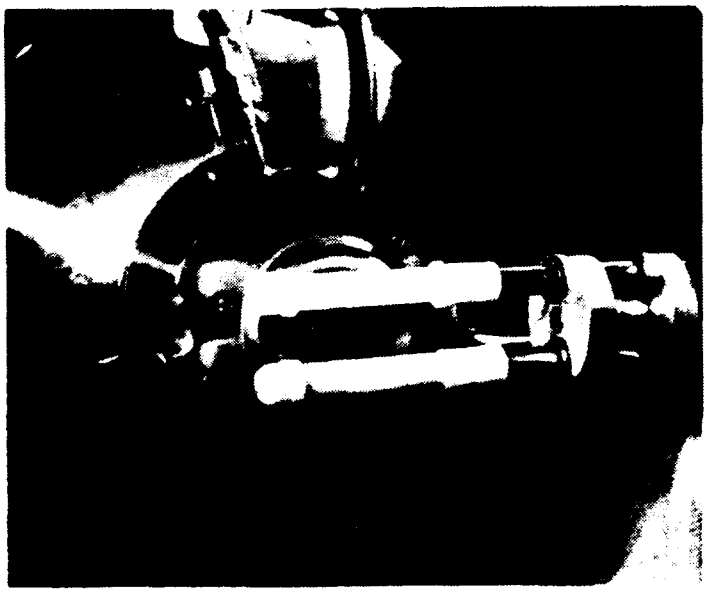

Fig. 3.

separate polyethylene holders for irradiation. In order to obtain a uniform neutron flux both sample and standard underwent three motions; rotation, revolution and traverse[5] as shown in Fig. 3. After irradiation for 20 sec the capsules were ejected from the neutron generator to the counting station /Fig. 4/ through a pair of flexible polyethylene tubes /3/4" diameter/ by means of compressed air. The samples were removed from the polyethylene holders by a specially designed stripper and dropped between two NaI/Tl/ detectors where a photocell was activated to start the counting sequence[9]. Samples and standard were sequentially dropped into the counting position. The complete irradiation and counting process took only 2 min. Such rapid and consistent transport enhances the accuracy of the oxygen analyses.

A 6.1 MeV gamma-ray is produced when ^{16}O reacts with a neutron to form 7.1 sec ^{16}N. As can be seen

Fig. 4.

from Table 1, the short-lived ^{16}N can be analyzed as many times as necessary without any interference from yttrium or aluminum.

The counting system consisted of two 5"x5" sodium iodide thallium crystals. A vertical adjustment system ensures that the sample is centered between the crystals to maximize the 4π geometry. The electronics of the counting equipment are shown in block diagram of Fig. 5. This system allows an energy window to be set up using a single channel analyzer. The counts in the energy window are used for the analytical data. By using a calibrated pulser any drift in the energy peak can be detected and adjusted.

From the radioactive decay equation it is seen that the rate of disintegration depends on the number of radioactive nuclei present at any time. Integration of the relationship.

TABLE 1

Nuclear reactions

Element	Reaction	Isotopic abundance, %	Cross section, m.barn	Half-life	Energy, MeV
O	$^{16}O/n,p/^{16}N$	99.76	42	7.13 s	6.13
					7.12
Al	$^{27}Al/n,p/^{27}Mg$	100	65	9.46 m	0.844
	$^{27}Al/n,\gamma/^{28}Al$	100	230	2.24 m	1.779
	$^{27}Al/n,\alpha/^{24}Na$	100	118	14.96 h	1.369
					2.754
Y	$^{89}Y/n,2n/^{88}Y$	100	600	106.6 d	1.836
Y	$^{89}Y/n,p/^{89}Sr$	100		50.6 d	none

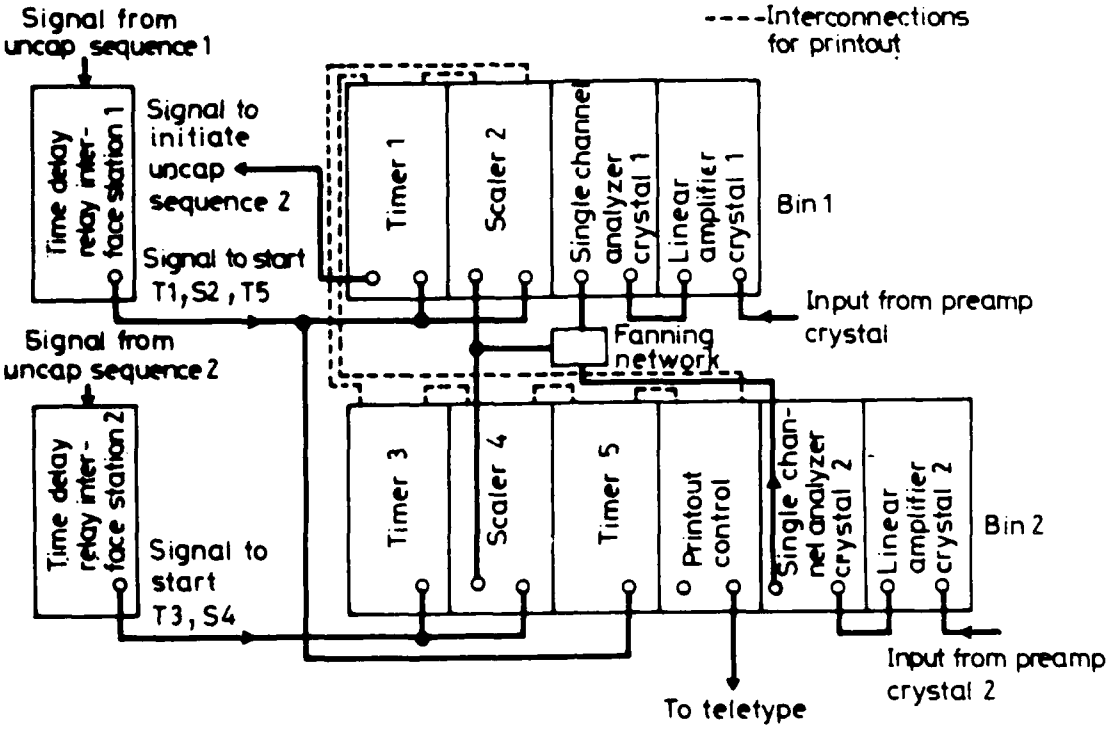

Fig. 5.

$$\frac{dN}{dt} = -\lambda N$$

gives $N = N_o e^{-\lambda t}$

where N = number of radioactive atoms at any time,

N_o = number of radioactive atoms at t=0,

λ = radioactive desintegration constant = $0.693/t_{1/2}$.

N_o is calculated for both the unknown and the standard. The counts in each counting interval is a definite integral.

$$N_o = \frac{N}{e^{-\lambda t}}$$

$$\int_{ta}^{tb} e^{-\lambda t}dt = \frac{e^{-\lambda tb}}{-\lambda} - \frac{e^{-\lambda ta}}{-\lambda}$$

$$N_o/unk/ = \frac{N/unk/}{\frac{e^{-\lambda t/unk/}}{-\lambda} - \frac{e^{-\lambda t/std/}}{-\lambda}}$$

$$N_o/std/ = \frac{N/std/}{\frac{e^{-\lambda t/std/}}{-\lambda} - \frac{e^{-\lambda/std/}}{-\lambda}}$$

The ratio of $N_o(unk)/N_o(std)$ is the ratio of oxygen in the standard and the unknown, and

$$\text{Oxygen/g/in unk} = \frac{N_o unk}{N_o std} \times \text{oxygen/g/in std.}$$

This method of calculation does not depend on the exact reproducibility of time, only the interval of time measurement. The sensitivity using this system is of the order of 1200-1300 counts per milligram of oxygen. The accuracy of this method is considerably enhanced by the removal of the irradiated capsule before counting.

Standard

High purity Al_2O_3, 99.992%, from Adolf Mueller Co., and Y_2O_3, 99.999% pure, in powder form from Moly Corp., were vibrated and tamped into a weighed polystyrene capsule. A 0.304" diameter x 1.250" long piece of Lucite was used for the oxygen standard. Once it was established that the YAG crystal was stoichiometric it was used as the standard. This simplified the analysis considerably.

Apparatus

A Kaman A-711 neutron generator was employed in this work. High energy neutrons were produced by the $^{3}H/d,n/^{4}He$ reaction using a 7 Ci tritium on a titanium target. The neutron flux was $5x10^{9}$ n $cm^{-2}sec^{-1}$ at the sample position with deuteron beam intensity of 4 mA.

RESULTS AND DISCUSSION

The quantitative results for yttrium and aluminum were based on the measurement of the activity of the 1.84 MeV gamma peak of ^{88}Y and the 1.369 MeV gamma peak for aluminum for both sample and standard. A simple comparison between sample count and standard count yielded the quantitative result. The values of these determinations are given in Table 2. As can be seen the relative standard deviation falls well within the quantitative region.

Sources of error resulting from variation of the neutron flux[9] from the generator, counting loss, and decay correction due to multichannel dead time and pulse pile up losses have been taken into consideration and eliminated.

CONCLUSION

This neutron activation technique is a powerful and useful tool and also unique for the stoichiometric analysis of YAG because it is nondestructive and allows macro-concentrations of three elements to be determined. There is no other analytical method that can accomplish this. In our particular experiments we had several

TABLE 2

Elemental analysis of YAG /wt %/

Element	Calculated theoretical	*No. of crystal	Content% mean + Std. dev. found
Yttrium	44.93	1	44.62+0.41
		2	44.84+0.39
		3	44.73+0.44
Aluminum	22.73	1	22.61+0.24
		2	22.72+0.21
		3	22.63+0.25
Oxygen**	32.34	1	32.12+0.16
		2	32.22+0.12
		3	32.26+0.21

*At least 10,000 counts were accumulated for each isotope.

**Each sample analyzed 5 times.

grams of sample. However, the technique is not concentration dependent as are most wet chemical methods.

Recently strong luminescence with a wavelength at 390 nm was discovered in YAG undoped single crystals[10]. Oxygen deficiency is suspected to be a cause of this fluorescence. By using the neutron activation analysis technique in determination of oxygen concentration in YAG can prove invaluable.

A value of $Y_3Al_5O_{12}$ was obtained from each of the three YAG samples indicating proper premixing before the furnace treatment. It was also found that a pure YAG crystal can be used as a comparative standard in the analysis of newly prepared YAG crystals.

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CHARACTERIZATION OF YTTRIUM ALUMINUM GARNET /YAG/ USING 14 MeV NEUTRON ACTIVATION - Forrest Burns, Michaeline F. Chen and Jaroslav L. Caslavsky

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